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METHODS FOR STUDYING SINGLE-CRYSTAL ALUMINUM-OXIDE FIBERS

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The central avenues for studying the fine structure of single-crystal α-Al_2O_3 fibers obtained by pulling from melt onto a seed (EFG) and with thinning of the feeder by laser radiation (LHPG) are presented. The crystallographic orientation of single-crystal α-Al_2O_3 fibers was investigated using optical and scanning electron microscopes. It is suggested that x-ray diffraction topography be used to study the degree of perfection of single-crystal α-Al_2O_3 fibers and to observe the boundaries of blocks and individual dislocations.

Key words: single-crystal α-Al_2O_3 fiber, EFG method, LHPG method, laser heating, pulling from melt, IR-laser fiber optics, x-ray microscopy.

Increasing the admissible working temperatures of propulsion units in the aviation industry and developing a new-generation high-load fiber optics are real problems whose solution will greatly increase the effectiveness of existing systems and in principle make it possible to develop new ones [1, 2]. The modern composite materials must be used in aviation and at higher operating temperatures in order to decrease fuel consumption, increase efficiency and decrease harmful emissions and, in the case of space vehicles, to reduce weight and increase payloads [3, 4]. Single-crystal α-Al_2O_3 oxide fibers can be used as an optical guide for high-energy IR-laser radiation [4]. Sapphire fibers for use in fiber optics have been studied the most, because the losses in IR-radiation transfer are low (< 0.4 dB/m for 300-μm fibers) [9, 10]. The stringent quality specifications for the fibers obtained make it necessary to study the fine structure of the fibers, which determines their ultimate macro properties.

The most suitable method for obtaining single-crystal fibers with a uniform diameter is pulling single crystals from melt onto a seed in definite temperature regimes, specifically, with a definite temperature gradient at the melt boundary. There are two variants of this method depending on the method of heating.

The first method is known in the literature as Edge-defined Film-fed Growth (EFG) or Stepanov's method [5, 6] (Fig. 1). In this method a single-crystal fiber is pulled from aluminum oxide melt from several dies simultaneously onto a single-crystal seed [5, 6, 8, 9]. The melt, which is located in a molybdenum crucible, flows upward along capillaries, whence a fiber grows onto a single-crystal seed. This method has been perfected in different countries, including Russia [6, 7]. The physical essence of this method consists in feeding melt from the crucible through the capillary channels onto the working surface of the die, where meshing with the external edges occurred. As the crystal is pulled, capillary forces, which move the melt from the crucible to the end of the die, continually fill the meniscus is. All this makes it possible to obtain single crystals with any prescribed shape.

The second method, known as Laser Heated Pedestal Growth (LHPG), implements the principle of laser heating of

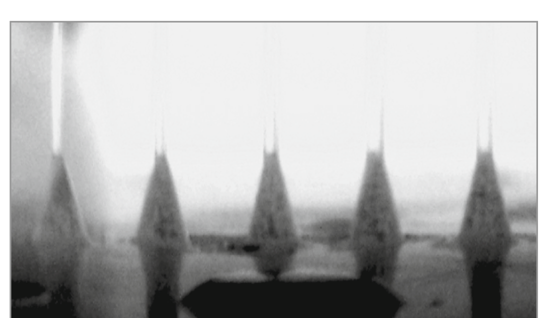

Fig. 1. Sapphire fibers no larger than 180 μm in diameter growing in a group regime.

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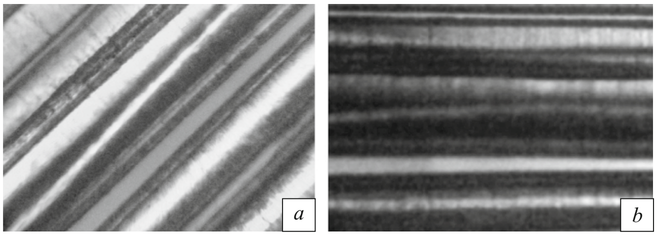

Fig. 2. Sapphire fibers, photographed in polarized light.

the molten zone located at the end of a feeding bar [7, 8]. The fiber grows in a special chamber, and the laser beam is transformed by means of an optical system in a manner so that its intensity is uniformly distributed in the form of a narrow ring over the surface of the seed, thereby ensuring local heating of the feeder (in contrast to a primary beam with the standard Gaussian distribution).

X-ray structural methods of analysis and observation of fibers in polarized light show that the fibers obtained by Stepanov's method have a single-crystal structure, and the orientation set by the seed crystal is preserved along the entire length of the grown fiber [8, 9]. In polarized light total extinction is observed with the angle changing away from 45° (Fig. 2).

The fracture surface of sapphire fibers grown along the [0001] axis is shown in Fig. 3*a*. A planar rhombohedron is observed on the surface; the angle between the normal to the face of the rhombohedron and the C axis is 57.6°.

An enlarged fragment of the surface of a fiber is shown in Fig. 4. Low fiber roughness of the fiber is achieved by means of the fiber stabilization system used to obtain fibers by Stepanov's method.

X-ray microscopy (diffraction topography) occupies a special place among the nondestructive methods used to study the structure of crystals [9]. The high sensitivity to lattice imperfections, which makes it possible to study boundaries, microcracks, dislocations and segregations of impurities, makes it possible to use this method to study single-crystal α-Al_2O_3 fibers.

X-ray topography can be used to determine, among other things, the type and spatial arrangement of dislocations in the interior of a single crystal by means of transmission topograms obtained from two mutually perpendicular projections. Together with dislocations it is possible to observe the block structure of a single crystal, stacking faults, twin boundaries and growth layers, which are due to the nonuniform distribution distribution in the process of crystal growth, as well as clusters of point defects. The character of the distortions of the crystal lattice can be determined by analyzing the contrast extinctions accompanying reflections from different types of planes. The spatial resolution of x-ray topography in the high-resolution Laue method (Fujiwara), presented in Fig. 5, is approximately 1.5 μm.

Fujiwara's transmission method is distinguished by the use of a microfocal source of white x-ray radiation. Photographing is done simultaneously in the diffracted beams reflected from different families of atomic planes of the single

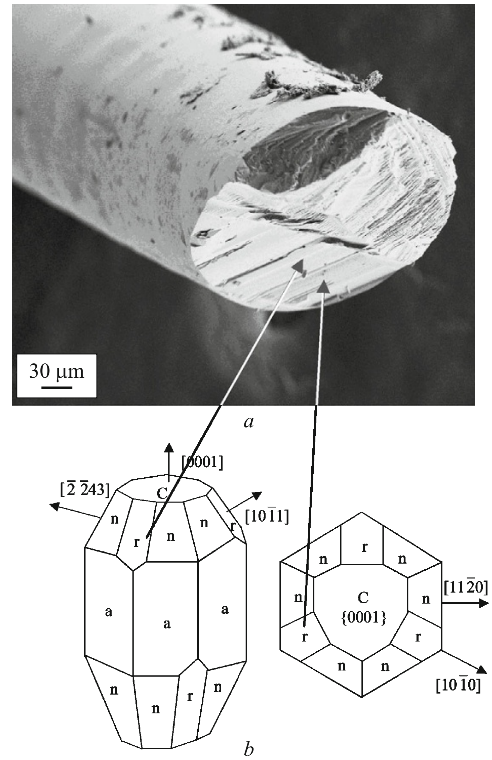

Fig. 3. Sapphire fiber grown along the [0001] axis.

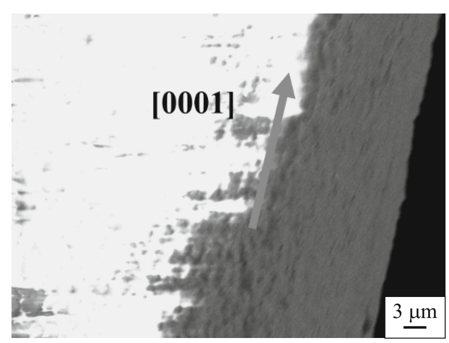

Fig. 4. Lateral surface of a fragment of a single-crystal fiber (the arrow indicates the orientation of the fiber).

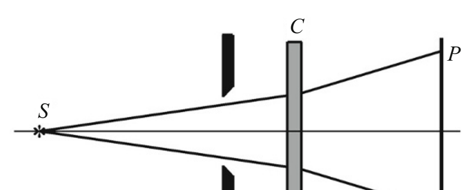

Fig. 5. Arrangment used in Fujiwara's transmission method: *S*) microfocal source of white x-ray radiation; *C*) experimental single crystal; *P*) photographic film.

crystal. Fujiwara's scheme is similar to the scheme used to obtain Laue photographs, but diverging beams of large area are used and the intensity distribution (image) in each diffracted beam is studied. Block boundaries and individual dislocations are visible in Fujiwara's x-ray topograms.

CONCLUSIONS

Avenues were shown for studying the fine structure of single-crystal α-Al_2O_3 fibers obtained directly by pulling from melt to a seed and by laser heating to thin the feeder.

The results of investigations performed on single-crystal α-Al_2O_3 fibers in polarized light under an optical microscope to study the single-crystal structure and the preservation of the orientation set by a seed single crystal.

The crystallographic orientation of the fracture surface of sapphire single-crystal α-Al_2O_3 fibers was studied.

It was proposed that x-ray diffraction topography be used to study the degree of perfection of single-crystal α-Al_2O_3 fibers and to observe block boundaries and individual dislocations.

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